

Research paper

Rutting Performance of Nano-Silica Modified C320 Bitumen: A Case study

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Abstract

Nanomaterials exhibit novel properties and profound attributes as an additive in asphalt binder modification. However, the application of nano-silica in asphalt binders and mixture modification is still limited and further research is required. Along these lines, in this work, nano-silica with a content from 2% to 8% and an increment of 2% were utilized in modifying the bitumen binder type C320, which is considered the most conventional type of bitumen used in Western Australia road asphalt mixtures. Various tests were performed to assess their properties including dynamic shear rheometer, penetration, softening point, and multiple stress creep recovery (MSCR) test. The extracted results revealed an increase in the strength and stiffness properties by lowering the penetration, improving the softening point, and increasing the complex shear modulus of all nano-silica modified bitumen samples. Interestingly, a relatively big content of nano-silica leads to a higher rutting resistance. Nevertheless, the rutting resistance was affected by the size of the nano-silica coated with the silane coupling agent. The ideal sample of nano-silica modified C320 was determined as NS-15nm (NS-A), which can improve the rutting resistance. Nevertheless, the rutting resistance of the modified asphalt mixtures needs to be further investigated in the future to elaborate on the impact of nano-silica as modified binders on the mechanical properties of Australian asphalt mixtures.

Key words: nano-silica, C320 bitumen, rutting, penetration, DSR, MSCR

1. Introduction

Road pavements start experiencing functional deterioration once they are open to heavy traffic. One way to increase the service life of road surfaces is using certain additives such as polymers to modify and improve the properties of the asphalt mixtures [1,2]. Polymer-modified binder mixtures have been widely used in various civil engineering and construction projects. The addition of polymers within these mixtures improves their stiffness and significantly enhances their robustness against temperature fluctuations. This modification, in turn, improves the mixture's resistance to pavement cracking. In addition, the incorporation of polymers into the binder results in a significant increase in its cohesiveness and adhesiveness, allowing it to effectively bind the mixture of components together [2]. However, the employment of polymers for bitumen modification has certain disadvantages in terms of storage stability, bitumen–polymer phase separation, and elevated costs [3]. Therefore, the scientific community aspires to find an alternative additive that could improve the bitumen binder's properties. From this perspective, nanomaterials are currently employed to adjust the characteristics of bitumen binder and asphalt mixtures. The superior surface area and effective particles network created in the modified bitumen increase their stiffness and improve the asphalt mixtures' resistance to permanent deformation [4,5].

Nano-silica has several applications in the medical and engineering sectors. As far as the building material and concrete industry is concerned, nano-silica shows an essential part in the adhesion and cohesion of concrete. In road and pavement materials, nano-silica exhibits a promising potential for improving asphalt mixtures' the mechanical and engineering properties. It can also be used in bitumen and asphalt modification owing to its several advantages, including low-cost production and practical characteristics [4]. Nano-silica is a

novel material that has many desirable characteristics such as a high surface area, enhanced absorption, high allocation, good stability, high purity, and is cost-effective by nature [4]. In section 2, the application of nano-silica in bitumen modification and the performance properties will be thoroughly illustrated and discussed.

To the best of the author's knowledge, nano-silica has not been used to modify the structural properties of bitumen C320, which is the most common bitumen used to design wearing course materials in Australia. Under this direction, the goal of this work is to investigate the rheological-physical properties and rutting resistance of nano-silica within the C320 bitumen and establish the ideal type of nano-silica to be used as an additive in the future nano-silica modified asphalt mixtures.

2. Nano-silica in bitumen modification

Nanomaterials including nano-silica display unique characteristics in comparison with the conventional material configurations due to their small size and high surface to volume ratio. Consequently, nano-silica holds crucial features, which can be employed in asphalt pavement as an additive [6,7]. The outstanding characteristics of nanomaterials such as their bulky surface area and self-assembly properties, which are quite different from the majority of the commonly used materials in construction, render them an idealistic material as additive for asphalt pavement applications. In addition, nanomaterials possess tremendous abilities that include self-cleaning and self-healing abilities [4,6,7]. These fundamental properties of nanomaterials successfully meet the requirements and standards of modern highway pavement. Therefore, nanoparticles have been already used to enhance the efficiency of asphalt [7].

Xiao and his associates' incorporated nanoparticles within asphalt to examine their rheological properties. In the last decade, numerous types of nanoparticles have been utilized to enhance asphalt's properties [8,9,10].

Several reports in the literature [7,8,9,11- 17] have demonstrated that nanomaterials can significantly enhance the cohesion of asphalt mixture and establish an enhanced linking between the nano-silica and asphalt, preventing thus the growth of cracks. As a result, the fatigue life would possibly be expanded, and the rutting failure substantially is declined [11-15] Various studies have been also conducted by using different contents and sizes of nano-silica and different mixing conditions, as illustrated in Tables 1-3.

As can be seen from Tables 1 and 3, the highest nano-silica content was up to the value of 6%, which can significantly improve the rutting resistance. The applied ideal mixing conditions were 2 hours, at 160 °C by using the high shear mixer of 4000 rpm, as illustrated in Table 3.

Table 1: Implementation of nano-silica in asphalt.

Reference	Asphalt	Nano-silica (%)
[11]	60/80	1.86-1.98%
[13]	PG-76	2%, 4%
[14]	PG 58-34	4% and 6%
[15]	60/70	2%, 4% and 6%
[18]	AH-70	3%, 5% and 7%

Table 2: Specification of the Nano silica properties based on previous studies.

Reference	Nano silica purity (%)	Size/dia. (nm)	Surface area (m ² /g)	Density g/cm ³
[4]	SiO ₂ > 99%	10nm	600	2.4
[5]	SiO ₂ > 99	11-13	200	2.4
[19]	SiO ₂ +carbon nanotube >95%	10-12	Not Given	2.64
[20]	SiO ₂ > 99	Not Given	195	Not Given

Table 3: Mixing aspects and mixing conditions of producing Nano silica.

Authors	% Nano-silica	Time	Temperature	Speed
[4]	2%,4%,6%, 8%	2 hours	135°C	4000 rpm
[5]	1, 3, and 5 %	1 hour	160°C	3000 rpm
[19]	1%, 3%, 5%	2 hours	160°C	4000 rpm

On top of that, the rutting and fatigue resistance of nano-silica has been systematically investigated [13-17] and the published results have indicated the remarkable ability of nano-silica to enhance the bitumen's rheology and mixtures mechanical properties. According to the literature [13-15], the unique features of nano-silica have considerably contributed to the tremendous enhancement of the modified bitumen properties. These features include extraordinary chemical purity, excellent dispersal skill, adsorption, and outstanding stability. In addition, various works focus on the physical-rheological properties of nano-carbon modified asphalt binder, [4,9,10] while less attention has been given to the modification of the asphalt using nano-silica. Yao et al [14] suggested that both chemical reactions and physical dispersion are possible to occur during the blending of nano-silica and bitumen, which could lead to the production of a new network structure. Therefore, further studies are required to shed light on these effects.

The performance properties, in terms of improved stiffness and less sustainability to moisture damage, have been previously investigated [13,15, 18]. In addition, the use of nano-silica with virgin polymer in bitumen modification, in terms of improving the physical and rheological properties have been also examined. From the reported outcomes, it can be argued that the reaction between nano-silica and bitumen is not a trivial issue. Thus, further research is required to examine the underlying origins of the above-mentioned interactions.

3. Experimental program

3.1 Materials

3.1.1. Bitumen

As far as binder selection is concerned, C320 bitumen was employed and supplied by SAMI Bitumen Technologies, which is in Perth city, Western Australia. This type of bitumen binder was selected following the specification and the recommendation of Main Road Australia and the Australian Asphalt Pavement Association, which both recommended the use of a C320 binder in wearing course materials design. Table 4 demonstrates the bitumen's physical properties [21].

Table 4: Bitumen's properties.

Properties	value	Australian Standards
Penetration at 25 °C	42 mm	A2341.12
Brookfield Viscosity at 135 °C	0.502 Pa.s	AS 2341.2
Flash point	248 °C	AS 2341.14

3.1.2. Nano-silica

In this work, three different sizes of nano-silica were used, as can be ascertained from the provided Scanning Electron Microscopy (SEM) image in Figure 1. Generally, three types of nano-silica have been used in this work, two of which were coated with a Silan coupling agent. The latter was used to improve the compatibility between the nano-silica and bitumen binder and to modify the silica's surface. Table 5 illustrates the properties of the three different types of nano-silica. NS-A stands for SiO₂, with a thickness value of 15 nm, coated with 2wt% KH550-Silane Coupling Agent. NS-B represents the nano-silica SiO₂, 99+%, with a thickness of 20-30 nm (no coating agent). NS-C denotes the nano-silica SiO₂, with a thickness of 30 nm coated with 3-4wt% KH550-Silane Coupling Agent.

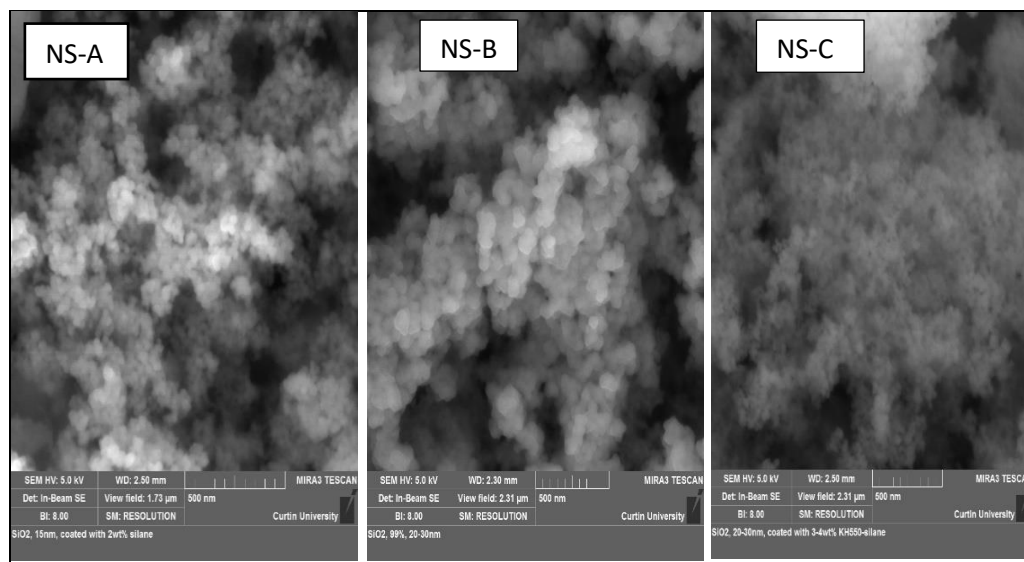


Figure 1: SEM images of the different types of nano-silica.

Table 5: Properties of different size of nano-silica

Nano name/	Sample name	area	size	colour	True density	Purity
SiO ₂ , 15 nm, coated with 2wt% KH550-Silane Coupling Agent.	NS-A	600 m ² /g	15 nm	White powder	2.4 g/cm ³	97.3 %
SiO ₂ , 99+%, 20-30 nm (no coating agent)	NS-B	180-600 m ² /g	20-30 nm	White powder	2.4 g/cm ³	99%
SiO ₂ , 30 nm coated with 3-4wt% KH550-Silane Coupling Agent.	NS-C	130-600m ² /g	30 nm	White powder	2.4 g/cm ³	96.3%

3.2 Sample preparations

The high-shear mixer type Silverson L5M-A was used to create nano-silica-modified bitumen structures with a variety of contents: 0%, 2%, 4%, 6%, and 8% by weight of bitumen for each sample of NS-A, NS-B, and NS-C, which refer to the sorts of nano-silica used, as is illustrated in Table 5. The mixing conditions included a high shear speed of 4000 rpm, a mixing time of 2 hours, and a processing temperature of $170\text{ }^{\circ}\text{C} \pm 0.5^{\circ}$. The mixing process started at 700 rpm for the first 10 minutes and then the speed was increased to 4000 rpm. The prepared samples of the modified bitumen were stored in a 1 L container, which were used for testing the physical, viscoelastic, and rutting properties.

3.3 Testing methods

3.3.1 Penetration test

The penetration test is considered a well-established method to determine the physical properties of the bitumen binder. The Australian Standard AS 2341.12 has been followed during this method. The penetration test conditions were $25\text{ }^{\circ}\text{C}$, 5 seconds and enforcement of 100 g load for measuring the vertical penetration distance of standard needle in the bitumen samples. The major outcome of the test was to determine the strength of bitumen and the capacity to resist rutting deformation.

3.3.2 Softening point test

The softening point test, which is also known as the ring and ball test, was used to determine the softening and stiffness levels of the bitumen. This approach can be used as an indicator of the bitumen's susceptibility to high temperature, whereas valuable insights regarding the lower temperature susceptibility and improved rutting resistance can be derived. The test was conducted according to the Australian Standard AS 2341.18 at $25\text{ }^{\circ}\text{C}$.

3.3.3 Dynamic shear rheometer (DSR)

This test was conducted according to the AASHTO 315 standards. This test aims to determine the impact of the complex shear stress and linear viscoelastic to the elastic properties of bitumen through oscillatory shear. More specifically, the primary objective of this test procedure was to determine the stiffness, elasticity and rutting resistance.

3.3.4 Multiple stress creep recovery (MSCR) test

The MSCR test was performed following the AASHTO T350-14 specification at 64 °C by using the DSR technique. The creep recovery and stiffness performance of the bitumen binder are determined, as well as the rutting resistance. The samples were tested in creep and recovery states under the application of stress loads of 0.1 and 3.2 kPa. The main two tested parameters of J_{nr} and %R represent the non-recoverable creep and recovery percentage.

4. Results and discussion

4.1 Physical properties

Figure 2 displays the penetration results of nano-silica at 25 °C. It is interesting to notice that the addition of different sizes of nano-silica indicates a considerable improvement in the penetration results for all contents of nano-silica. However, NS-A exhibits better results at 6–8%, which could be attributed to the impact of small size nano-silica (15 nm) and the use of KH550-Silane as the coupling agent. The use of KH550-Silane can significantly improve the compatibility and interaction between the bitumen and nano-silica because [7,17].

In addition, a similar trend was observed, in terms of the softening point, as is shown in Figure 3. The optimum physical properties could be achieved by using NS-A, which is

nano-silica with a size of 15 nm coated with the coupling agent. However, all nano-silica modified samples have higher softening points than the non-modified C320 bitumen. The finding is in line with the previously reported findings in the literature [12,15]. However, in the current study by using the 6-8% of NS-A, a relatively high improvement in the penetration and softening properties up to 45% was achieved compared to the non-modified binders.

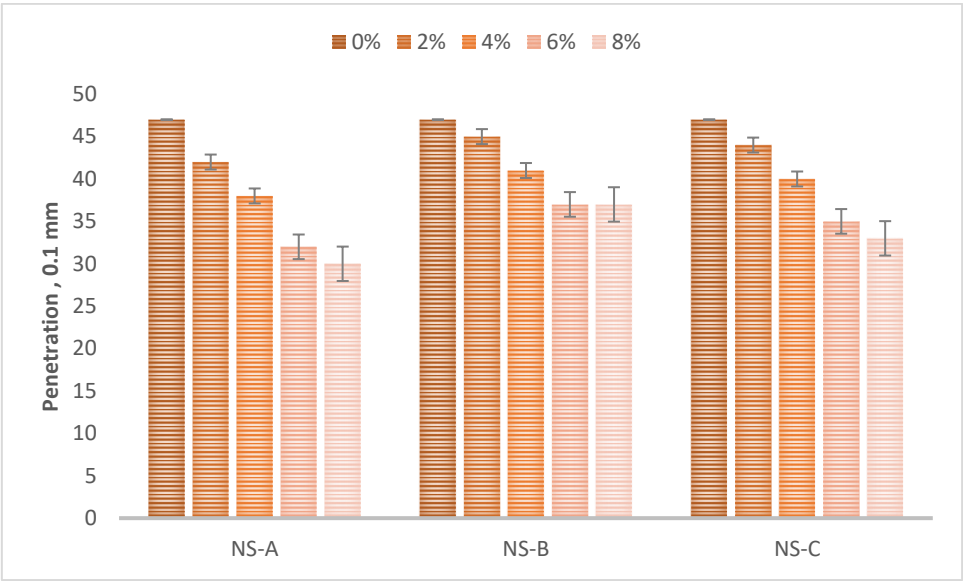


Figure 2: Penetration results.

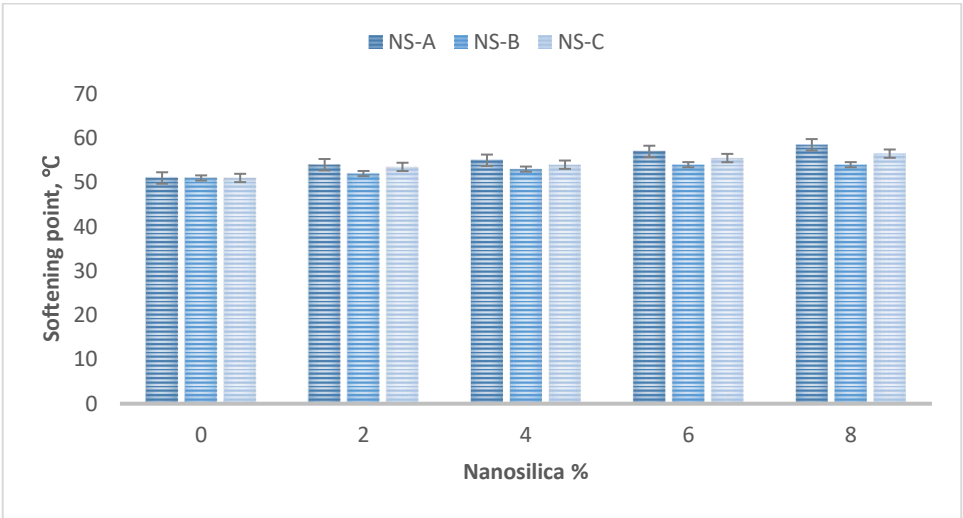


Figure 3: Softening point results

4.2 Rheological properties of nano-silica modified C320 bitumen

Tables 6-8 illustrate the stiffness properties, in terms of the complex shear modulus for all the different types of nano-silica. By using various temperatures of 50 °C, 58 °C, 60 °C, 64 °C, 70 °C, and 76 °C. Tables 6-8 illustrate the influence of nano-silica percentage, size, and types on the complex shear modulus of the modified bitumen. Results show that different size and different content of nano-silica would significantly improve the stiffness properties by increasing the complex shear modulus. Therefore, better stability and high resistance to deformation. Thus, nano-silica modified C320 bitumen results in an enhancement in rutting resistance. Modifying and advancing the properties of bitumen and asphalt mix by using certain additives, such as nano-silica, is one way of boosting the service life of road surfaces [4,19].

Table 6: Complex shear modulus of NS-A.

	50°C	58 °C	60 °C	64 °C	70 °C	76 °C
0%	14.2	3.87	3.2	2.76	0.8	0.4
4%	15.89	7.6	5.4	2.9	1.9	0.42
6%	16.66	5.9	4.3	2.5	2.1	0.5
8%	18.6	7.1	4.8	4.2	2.78	0.5

Table 7: Complex shear modulus of NS-B.

	50°C	58 °C	60 °C	64 °C	70 °C	76 °C
0%	14.2	3.87	3.2	2.76	0.8	0.4
4%	14.89	5.6	4.4	1.9	1.9	0.42
6%	16.66	5.9	4.3	1.5	2.1	0.5
8%	18.6	9.1	5.8	1.2	0.78	0.15

Table 8: Complex shear modulus of NS-C.

	50°C	58 °C	60 °C	64 °C	70 °C	76 °C
0%	14.2	3.87	3.2	1.76	0.8	0.4
4%	16.89	5.6	4.4	2.9	1.9	0.42
6%	16.66	4.9	3.3	2.5	2.1	0.5
8%	19.6	6.1	4.8	3.2	2.78	0.5

4.3 DSR Rutting of nano-silica modified C320 bitumen

The rutting factor ($G^*/\sin \delta$) has been used as a rutting indicator and the extracted results of the various samples modified with different nano-silica sizes are shown in Figure 4. At the temperature value of 64 °C, the 8% NS-A and NS-C samples had a higher rutting resistance than the unmodified bitumen. More specifically, the rutting factors of the NS-A and NS-C samples were 7.1 kPa and 5.88 kPa, respectively, whereas the rutting factor of NS-B was only 2.45 kPa. Although both NS-B and NS-C samples had the same size of 20–30 nm, the implementation of a 2% coating of silane coupling on NS-C resulted in an increase in the rutting resistance in comparison to NS at a thickness value of 20–30 nm without incorporating a coupling agent coating.

Nanomaterial and nano-silica could substantially improve the adhesion and cohesion of bitumen binder and created a bridging impact between the bitumen and nanoparticle, evading the growth of deformation [15,17, 20]. Therefore, in turn, the life service of road pavement may possibly be extended, and the rutting failure significantly declined.

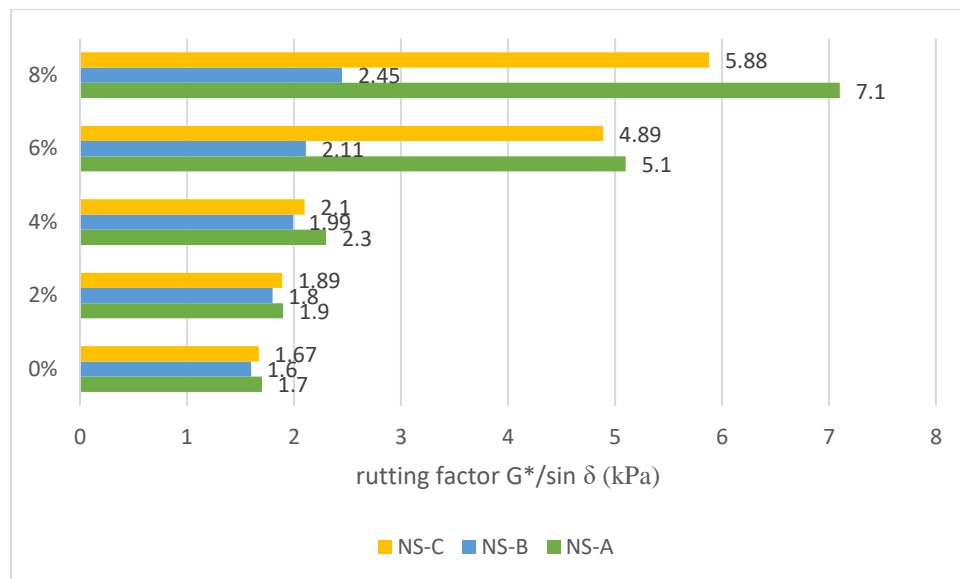


Figure 4: Rutting factor of nano-silica-modified bitumen

4.4 MSCR Rutting of nano-silica modified C320 bitumen

Figure 5 illustrated the recovery percentage of nano-silica-modified bitumen C320 and the non-modified bitumen with three different nano-silica sizes under the application of high and low-stress levels after rolling thin film oven test RTFOT ageing. This study is considered of great importance for evaluating the rutting deformation. From the acquired results shown in Figure 5, the incorporation of nano-silica as a modifier yields an increase in the elastic properties and the recovery performance of the modified bitumen at elevated temperatures. All nano-silica-modified bitumen structures possess a higher percentage of recovery in comparison to the conventional non-modified bitumen.

Based on the results of these experiments, adding the nano-silica significantly increased the rutting resistance of the C320 bitumen modified mixture. Moreover, using nano-silica as an additive to bitumen samples results confirmed that by addition of the nano-silica

results in improving the rutting resistance, workability, stiffness, and efficiency of the modified bitumen [4,12-16].

Figure 6 depicts the non-recovered strain for nano-silica-modified bitumen C320 and the non-modified bitumen with three different nano-silica sizes under the implementation of high and low-stress levels. Interestingly, most of the modified samples exhibit a decrease in J_{nr} at high-stress levels. Since non-recovered strain is considered a sensitivity indicator against rutting deformation, it is clear that – in comparison to the non-modified bitumen – all nano-silica modified bitumen samples do have a lower susceptibility for the accumulation of non-recoverable strain under creep loading and recovery [22,23].

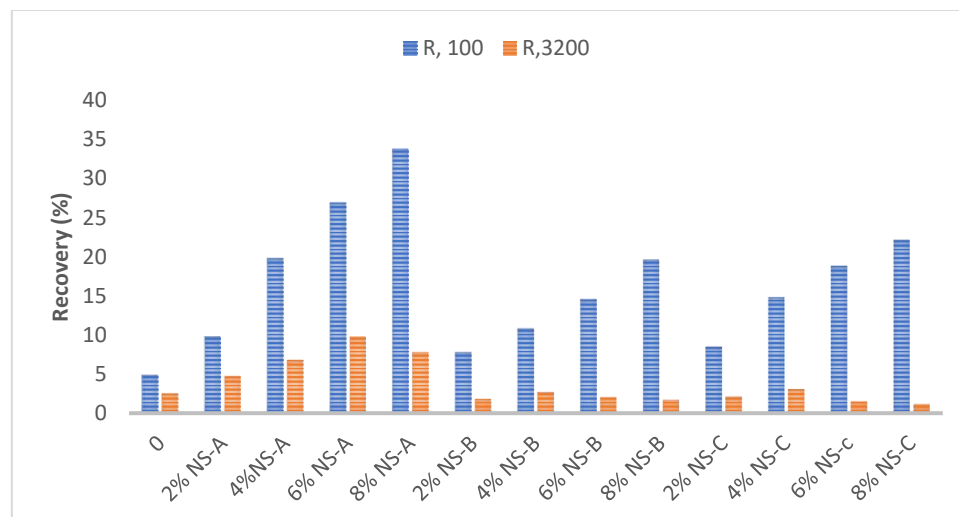


Figure 5: Recovery percentage of nano-silica-modified bitumen.

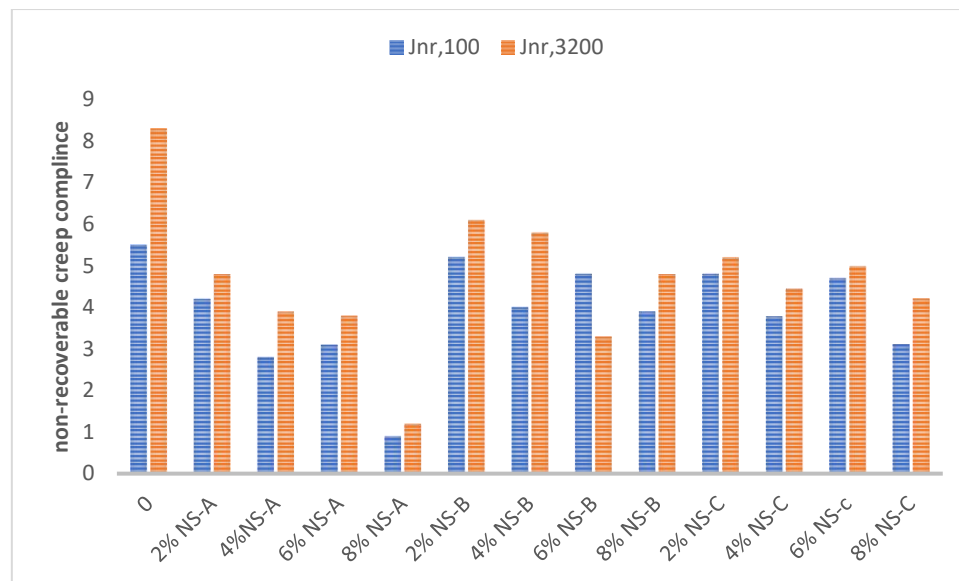


Figure 6: Non-recovered strain for nano-silica-modified bitumen

5. Conclusion

The employment of polymers for bitumen modification has some disadvantages, in terms of storage stability, bitumen–polymer phase separation, and elevated costs. Therefore, alternative additives should be found to improve the bitumen properties. Under this direction, nanomaterials and nanotechnology are currently employed to modify the structural properties of bitumen binders and asphalt mixtures. The findings of the study are as below:

- 1- The formation of a superior surface area and effective nano-bitumen-particle network in the modified bitumen can significantly improve the stiffness of the modified bitumen and enhance the asphalt mixtures' resistance to permanent deformation. Resulted show improvement in the resistance to temperature sustainability in terms of better penetration and softening points results of the modified samples.

- 2- From the extracted results, an increase in the stiffness properties was observed by lowering the penetration. Moreover, the mechanical strength was enhanced, in terms of the complex shear modulus of all nano-silica modified bitumen samples. The incorporation of a relatively high the percentage of nano-silica 6% and 8% led to a bigger rutting resistance. However, the trend followed by the rutting factor was not consistent, as the rutting resistance was also affected by the size of the nano-silica coated with the silane coupling agent.
- 3- The non-recovered strain for the nano-silica-modified bitumen C320 and the non-modified bitumen at three different nano-silica sizes for high and low-stress levels were found. Most modified samples exhibited a decrease in Jnr under the application of high-stress loads. Therefore, the utilization of NS-A leads to a reduction in the levels of rutting deformation.
- 4- Based on the results of this work, the implementation of NS of 15 nm coated with the silane coupling agent was selected for further research in the next work on producing the hybrid polymer-nano-silica-modified C320 bitumen. Several tests including rutting, durability and fatigue on both binders and asphalt mixtures will be conducted.

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Conflicts of Interest: The authors declare no conflict of interest.

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